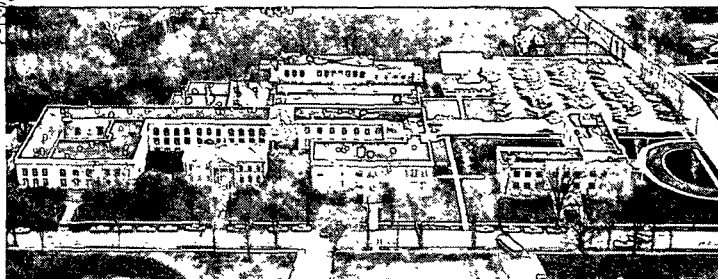


Property of
GEORGIA-PACIFIC CORPORATION
Technical Information Center
Atlanta, Georgia



THE INSTITUTE OF PAPER CHEMISTRY, APPLETON, WISCONSIN

**IPC TECHNICAL PAPER SERIES
NUMBER 166**

**THE USE OF MICROWAVE ATTENUATION AS A MEASURE OF
FIBER ORIENTATION ANISOTROPY**

C. C. HABEGER AND G. A. BAUM

APRIL, 1986

**THE USE OF MICROWAVE ATTENUATION AS A MEASURE OF
FIBER ORIENTATION ANISOTROPY**

Property of
GEORGIA-PACIFIC CORPORATION
Technical Information Center
Atlanta, Georgia

C. C. Habeger and G. A. Baum
The Institute of Paper Chemistry
P.O. Box 1039
Appleton, Wisconsin 54912

Abstract

A laboratory instrument which uses microwave attenuation to indicate fiber orientation anisotropy is described. The sensitivity of the instrument to fiber orientation, basis weight, furnish, moisture content, and density are investigated. The technique is recommended for on-line application if the results are corrected for varying moisture content.

Introduction

The distribution of fiber orientations in paper is primarily a function of conditions at the juncture of the pulp jet and the forming wire. The orientation distribution, which is skewed to the machine direction (MD), causes the physical properties of the paper to vary with angle to the MD. Fiber orientation anisotropy is not the only source of sheet anisotropy. Wet straining in the open draws and MD tension during drying also produce MD to CD (cross direction) differences in some properties. An on-line measurement of the anisotropy in a property, which is sensitive to fiber orientation (but not drying conditions), could be a monitor of pulp flow at the jet. A device which scanned the web in cross machine direction would be particularly useful, as it would detect sheet variability due to the inhomogeneous application of pulp to the wire.

The purpose of this paper is to describe a new instrument which indicates fiber orientation anisotropy; however, it begins with a brief discussion of the already existing methods. The distribution of fiber orientations was first measured on sheets formed from a pulp with a small portion of darkly colored fibers (1). The angles of orientation of thousands of dyed fiber segments were manually measured. Later, the angle tabulations were automated with a digitizer interfaced to a computer (2). This most direct fiber orientation determination has two limitations: (1) The sheet must have dyed fibers; and (2) if the sheet is not impregnated with a fluid whose index of refraction nearly equals that of the fibers, only dyed fibers near the surface are analyzed. Surface fiber orientation is often not representative of the entire sheet.

X-ray diffraction has also been used to find the fiber orientation distribution (3,4,5). The intensity of a Bragg diffraction peak is taken as a function of the angle from the line of intersection of the plane of radiation with the sheet to the machine direction. The axes of the cellulose crystals have a preferential alignment to the fiber axis. Therefore, the diffraction pattern of a fiber depends on the angle of the radiation to the fiber axis, and in turn the pattern of the sheet depends on the fiber alignment to the MD. This is a relatively rapid off-line measurement which can be applied to a wide range of basis weight. However, the distribution of fibril angle alignments to the fiber axis must be known to relate x-ray anisotropy to fiber orientation anisotropy.

The ratio of MD to CD zero span tensile strength is another laboratory measure of fiber orientation anisotropy (6,7). The zero span tensile strength (ZS) is the breaking load per unit width when paper is clamped between closely spaced jaws (8). When properly performed (9,10), it is a measure of fiber strength, which is insensitive to interfiber bonding. Since the fibers have greater tensile strengths along their axes, the ratio of MD to CD ZS can (for a given furnish) be an indication of fiber orientation anisotropy. Fiber strength increases with drying stress which is greatest in the MD. Thus, for the ZS anisotropy to only reflect fiber orientation anisotropy, sheets are first soaked in water for 24 hours to relieve drying stresses. The ZS test is an adequate test of fiber strength only for samples with basis weight of 30 to 80 g/m² (9).

Information about fiber orientation is also inferred from the pattern of visible light transmitted through a sheet. When laser light passes through a thin (< 50 μ m) sheet and is focused on the plane of a detector, light diffraction by single fibers creates an elliptical pattern. The principal axis of the

ellipse is aligned in the CD, and the ratio of signal when the detector is rotated along the CD to that along the MD is an indication of fiber orientation anisotropy (11,12). Apparently as the basis weight of the sheet increases, scattering (rather than diffraction) becomes the dominant mechanism in the creation of an anisotropic transmittance pattern. The major axis of the ellipse is now in the MD. Lippke manufactures both laboratory and on-line instruments (13) which use the scattering principle to predict fiber orientation anisotropy. Adequate light transmittance is achieved on sheets up to 250 g/m².

Background

The interaction of microwave radiation with paper is very sensitive to the moisture content of the paper. Water molecules have permanent dipole moments, which at low frequencies align with the electric field. The time required for dipole orientation is of the order of one cycle of the electric field at microwave frequencies. Therefore, the dielectric constant of water is larger (~80) below microwave frequencies than at higher frequencies (~5). In the microwave regime water dipole moments trail the electric field in time, and the imaginary part of the dielectric constant is large. Significant microwave energy is dissipated as heat. For sheets of moderate moisture content, water is the main source of labile dipoles and the dielectric constant of paper (especially the imaginary part) depends on the amount of water, its molecular environment, and the geometry of its distribution. Other sheet variables have only small influences on paper microwave directional properties (18). Microwave techniques for measuring moisture content rely on this strong interaction between water and microwave radiation.

The effective dielectric constant of paper, ϵ_p , depends on the orientation of the electric field with respect to the principal axes of the

paper. The absolute value of ϵ_p is greater for an in-plane than an out-of-plane electric field (14-17). Also, ϵ_p is larger for an electric field parallel to the MD than parallel to the CD (18,19). There are two explanations for this dielectric anisotropy (18). Paper is composed of fibers which lie in the plane of the paper and are preferentially aligned in the MD. This fiber level anisotropy in the geometry leads to dielectric anisotropy. The fiber fraction, which has a higher dielectric constant, is more connected along the machine direction and ϵ_p is thereby larger in the MD. Roughly speaking, the mixture is closer to a parallel alignment of capacitors in the MD. While the dielectric anisotropy can be explained in terms of fiber geometry, a secondary mechanism may also contribute. The bond between the absorbed water and the fiber restricts the lability of the water dipoles. On average, the water lability could be different along the fiber axis than transverse to the axis. This, along with the dielectric anisotropy of the dry fiber, could result in a fiber matrix that is not dielectrically isotropic. The anisotropy in the fiber would be reflected in the effective sheet properties. Regardless of the relative importance of the two mechanisms, sheet dielectric anisotropy will be sensitive to the fiber orientation distribution.

The change in in-plane dielectric constant with the angle of the electric field to MD is small; however, ϵ_p can be determined with sufficient repeatability to distinguish MD from CD and to detect changes in fiber orientation (18). Unlike mechanical anisotropy measurements, the ratio of MD to CD ϵ_p is insensitive to drying conditions (19). Therefore, microwave dielectric anisotropy could be a nondestructive indicator of in-plane fiber orientation anisotropy. There is much experience with on-line microwave moisture gages, and

development of an on-line fiber orientation indicator should be straightforward. However, before on-line work is justified a viable laboratory instrument must be developed and tested as a measure of fiber orientation and for sensitivity to sheet variables other than fiber orientation. The effective dielectric constant of paper is a well defined physical quantity whose direct measurement is tedious. A practical instrument for fiber orientation could determine an easily measured property related to ρ . It is the purpose of this paper to describe such a device and to discuss its benefits and limitations.

Experimental

The laboratory instrument described below provides a simple, rapid test of in-plane fiber orientation anisotropy based on microwave dielectric anisotropy. It measures the attenuation of a microwave signal passing through a sample. The attenuation depends on the amount of energy reflected at the sample-air boundaries and the amount of energy dissipated in the sample. These quantities, in turn, depend on the real and imaginary parts of the dielectric constant and the sample thickness. The effective dielectric constant is greater when the electric field is aligned in the MD; therefore, the ratio of attenuation with the electric field in the MD to that in the CD (R_M) is a measure of fiber orientation anisotropy. Briefly, the technique is to measure attenuations for a sample placed in a microwave waveguide at different orientations to the field. This is a simpler approach than finding the complex dielectric constant.

Figure 1 is a schematic diagram of the gage. The microwave signal is produced by a Polarad model 1108A-C X-band microwave signal generator having a frequency range of 8.2 to 12.4 GHz. All results reported were taken at 9.25 GHz. The microwave signal is pulsed on and off with 50% duty cycle at a 1 kHz rate. This amplitude modulated microwave signal goes through a Hewlett Packard 11686A

filter to eliminate spurious low frequency signals. The signal is next carried through a rigid coaxial cable to a rectangular X-band waveguide. The waveguide is 2.286 cm. wide and 1.016 cm. high. The electric field is oriented vertically (along the smaller dimension). The end of the waveguide is attached to a variable, microwave attenuator (Systron Donner DBG 430) with spring loaded bolts. The attenuator connects to a Systron Donner DBG 310 detector mount, which contains an Alpha Industries DDC4561D low barrier Schottky diode. The attenuator and detector mount are attached to a custom-made carrier which can be manually translated to open a gap between the waveguide and the attenuator. A switch shuts off the signal generator when the gap is open. Samples can be inserted in the gap with the MD vertical or horizontal. The signal, after passing through the sample and the attenuator, is rectified by the diode. The resulting 1 kHz square wave is applied to a Hewlett Packard 415E standing wave ratio meter. This is a narrow band amplifier centered at 1 kHz. It registers in decibels the incoming signal on a needle dial. Figure 2 is a photograph of the instrument.

(Fig 1 and 2 here)

One of our early difficulties was the lack of repeatability in the S.W.R. meter readings. These were sensitive to small movements of the signal generator and waveguides, to small movements of the then flexible coaxial cable into the waveguide, and to variability of the seating of the sample between the attenuator and the waveguide. This problem was remedied by bolting the signal generator, waveguide, attenuator, and detector mount to a common rigid base; replacing the flexible coaxial cable with a rigid one; and carefully aligning the translating carriage to avoid seating variabilities.

The first tests were conducted without the attenuator between the sample and the detector. This led to unsatisfactory results because of the large standing wave ratio in the microwave cavity. The loss was not linear with the number of samples inserted, and sometimes there were unreasonably high or low readings. The attenuator removed this problem by preferentially reducing multiple reflected components in the signal. A setting of 10dB was sufficient to avoid difficulties.

The procedure for determining the microwave anisotropy begins with the sample preparation. The sample is cut into strips 2.78 cm wide and about 15 cm long. This width is selected so that the sample will fit snugly between the bolts when placed in the waveguide. Strips cut along both the MD and CD are used. The strips are stacked to a thickness of about 0.15 cm and stapled at one end. Four small edge marks, about 2.5 cm apart, are made toward the middle of the stack. The data gathering procedure is to (1) pull open the gap, (2) insert a stack with the waveguide edge flush to an edge mark, (3) close the gap, (4) wait thirty seconds for the meter reading to stabilize, and (5) record the meter reading less the no sample value. This is done for each edge of the four marks with MD and CD stacks and with horizontal and vertical insertion. A total of sixteen readings are thereby taken. The first reading from the MD vertical test is added to first reading from the CD horizontal test. This quantity is divided by the sum of the other two first readings (CD vertical plus MD horizontal) to give a value of R_M . These calculations are carried out for each of the four edge marks and averaged to get R_M for the sample. The reason for using a relatively thick stack is to quickly average out sheet variability and to increase the signal-to-noise ratio in the attenuation reading. It is necessary to do

both vertical and horizontal insertions of MD and CD stacks, since the boundary conditions at the waveguide-detector mount interface are different for horizontal and vertical insertion.

Results and discussion

The first experiments investigated the sensitivity of R_M to fiber orientation changes. Sheets with four different fiber orientations were made in the laboratory using a Formette Dynamique (20). The common furnish was a predominantly Douglas-fir, bleached kraft, never dried pulp. Since the samples were fully constrained in the MD and CD while drying, the differences in anisotropy of the physical properties could be attributed to fiber orientation anisotropy only. Figure 3 is a plot of R_m vs. R_E , the ratio of the elastic stiffness (measured ultrasonically) in the MD to that in the CD. The error bars, which are typical for results reported later, are two standard deviations wide. Notice that although elastic anisotropies are larger, the ratio of uncertainty in a reading to variability between the samples is nearly identical in the mechanical and microwave tests. This indicates that the uncertainty in both cases is largely due to variability in the sample. Wet zero span anisotropies were also measured, and for this set of samples these values are very close to the elastic anisotropies. These results demonstrate that, on fully restrained sheets, R_M is an indicator of orientation anisotropy of equal quality to mechanical determinations. Further evidence of the utility of R_M comes from two measurements on commercial linerboard made at different rush-drag ratios and constant draw tension. The normal rush-drag setting produced an R_M of $1.056 \pm .006$ and an R_E of 2.27 ± 0.27 , where as a low rush-drag setting gave an R_M of $1.014 \pm .005$ and an R_E of 1.77 ± 0.21 .

(Fig. 3 here)

With the variable attenuator set at 10dB, R_M is not sensitive to basis weight. This statement is supported by the results in Table 1. Here R_M was determined on the same set of eight MD and eight CD liner board strips in three different ways. The sheets were inserted into the waveguide in stacks of two, four, or eight strips. These results also justify the choice of about 0.15 cm for a standard stack height. This is near the thickness of the four deep stack in Table 1. This is enough sample to minimize reading variability without losing basis weight variability at high gap spacings.

(Table 1 here)

One advantage of this technique is that the microwave attenuation is insensitive to thickness direction dependence in fiber orientation. To demonstrate this, an extremely two-sided stack was tested. The stack was made from MD and CD strips of the most oriented sample in Fig. 3. Eight MD strips were placed on the "top" side and eight CD on the "bottom." The stack was placed vertically into the waveguide and average attenuation was recorded with the top side toward the detector (2.443 dB) and the top side toward the generator (2.431 dB). The standard deviations in the readings were .021 dB and .023 dB, respectively.

The effect of furnish on the relationship between microwave anisotropy and elastic anisotropy was investigated by plotting R_M vs. R_E for Formette sheets of three different bleached kraft furnishes all dried under full restraint (see Fig. 4). The results indicate that, except at very high anisotropy, furnish has a similar effect on R_M and R_E .

(Fig. 4 here)

The sensitivity of R_M to moisture content is shown in Fig. 5. Measurements of R_M were taken at standard conditions (50% RH, 23°C) and then in a variable humidity room maintained at 15% RH or 75% RH. The calculated values of R_M are plotted vs. R_E measured at 50% RH. Notice that R_M increases significantly with moisture content.

(Fig. 5 here)

Sheet density can change R_M at constant fiber orientation. Table 2 demonstrates the relative effects of fiber orientation and wet pressing on R_M . Although R_M is much more sensitive to fiber orientation, there is a significant decrease in R_M (also in R_E) with density.

(Table 2 here)

A mechanism for the increase in R_M with moisture content can be visualized by considering an extreme case. Imagine a two-phase mixture with dielectric constants ϵ_F and ϵ_A , and let p represent the volume fraction of the F component. The geometry of the mixture is such that all interfaces are perpendicular to the y-axis. Therefore, the phases are aligned in series in the y-direction and in parallel in the x-direction. The ratio of the effective dielectric constant in the x-direction to that in the y-direction is:

$$R_I = (p\epsilon_F + (1-p)\epsilon_A) (p\epsilon_A + (1-p)\epsilon_F) / \epsilon_A\epsilon_F \quad (1)$$

The major dielectric effect of adding moisture to a sheet is to increase the dielectric constant of the fiber fraction. This is analogous to increasing ϵ_F in Eq. (1). Taking the derivative of Eq. (1) with respect to ϵ_F gives

$$\frac{\epsilon_F}{R_I} \frac{\partial R_I}{\partial \epsilon_F} = \frac{(1 - \rho) [\rho \epsilon_F^2 + 2(1 - \rho) \epsilon_F \epsilon_A - \rho \epsilon_A^2]}{(\rho \epsilon_F + (1 - \rho) \epsilon_A) (\rho \epsilon_A + (1 - \rho) \epsilon_F)} \quad (2)$$

This quantity lies between 0 (when ρ approaches one) and 2 (when ρ approaches zero). So, R_I rises with moisture content especially at low ρ . Increasing ϵ_F has a larger percentage effect on the dielectric constant in the parallel direction than in the series direction.

The effect of density could be investigated similarly by taking the derivative of Eq. (1) with respect to ρ . However, the result is that $\partial R_I / \partial \rho$ equals a positive number times $(1 - 2\rho)$. This conflicts with our experimental results which showed R_I decreasing as density increased even when the volume fraction was less than one half. The volume fraction was estimated by assuming that the fiber density was 1.5 g/cm^3 . The parallel-series model is probably inappropriate in this case, since it does not allow for increasing y-direction connectedness with increasing density. That is, as ρ increases the CD geometry becomes less series-like, and this oversimplified model does not account for that.

Conclusions

The microwave attenuation anisotropy ratio measurement described above is a simple laboratory indicator of fiber orientation. Testing should be done in a humidity controlled room to avoid the effects of changing moisture content. Also, caution should be taken when comparing results on sheets of greatly different densities.

The technique could be applied on-line to a wide range of basis weight sheets, provided the results are corrected for moisture. To our knowledge, the Lippke fiber scattering device is the only existing on-line fiber orientation indicator. A microwave meter could operate above the 250 gm/m^2 basis weight

limit of the Lippke device. Below this limit, it is hard to compare the two techniques, since no study of the sensitivity of the Lippke device to sheet variables other than fiber orientation has been published.

References

1. Danielsen, R., and Steinberg, B., Svensk Papperstid., 50:301(1947).
2. Crosby, C. M., Eusufzai, A. R., Mark, R. E., Perkins, R. W., Chang, J. S., and Upleskar, N. V., Tappi, 64(3):103(1981).
3. Prud'homme, R. W., Hein, N. W., Noach, J., and Marchessault, R. H., J. Applied Polymer Science, 19(9):2609(1975).
4. Koran, Z., Bussiers, D., and Prud'homme, R. E., Tappi, 65(8):88(1982).
5. Paakkari, T., Serimaa, R., Hattula, T., and Ahtee, M., Paperi Puu, 10:569 (1984).
6. Kallmes, O. J., Tappi, 52(3):482(1969).
7. Votava, R. J., Tappi 65(4):67(1982).
8. Van den Akker, J. A., Lathrop, A. L., Voelker, M. H., and Dearth, L. R., Tappi, 45(8):416(1958).
9. Wink, W. A., and Van Eperen, R. H., Tappi 45(1):10(1962).
10. Cowan, W. F., and Cowdrey, J. K., Tappi, 57(2):90(1974).
11. Rudstrom, L., and Sjolín, U., Svensk Papperstid. 73(5):117(1970).
12. Sadowski, J. W., Paperi Puu, 9:588(1979).
13. Kohl, A., and Hartig, W. Zellcheming general meeting. (Baden-Baden), 80th, June 24-28, 1985. 22 p.
14. Servant, R., and Gougeon, C. R., Acad. Sci. Paris 242:2318 (1956).
15. Chene, M., Coumes, A., and Lafaye, C. R., Acad. Sci. Paris 260(13):3632(1965).
16. M'Baye, K., and Pellisier, J. P., Rev. ATIP 29(2):51(1975).
17. Dusiou, N., Balaneschu, G., and Liuiu, T., Celul. Hirtie, 25(1):34(1976).
18. Habeger, C. C., and Baum, G. A., J. Appl. Polymer Sci., 28:969(1983).
19. Fleischman, E. H., Baum G. A., and Habeger, C. C., Tappi, 65(10):115(1982).
20. Sauret, G., Bull. ATIP, 1616:446(1962).

Table 1. Basis weight sensitivity

Sample	Microwave Anisotropy
4 each, 2 stack	1.070 ± 0.032
2 each, 4 stack	1.071 ± 0.007
1 each, 8 stack	1.063 ± 0.007

Table 2. Wet pressing effects

Orientation	Wet Pressing	Density, g/cm ³	Microwave Anisotropy	Elastic Anisotropy
Medium	High	0.827	1.093	2.28
Medium	Medium	0.656	1.127	2.55
Medium	Low	0.418	1.138	2.73
High	Medium	0.654	1.154	3.08
Low	Medium	0.621	1.008	1.17

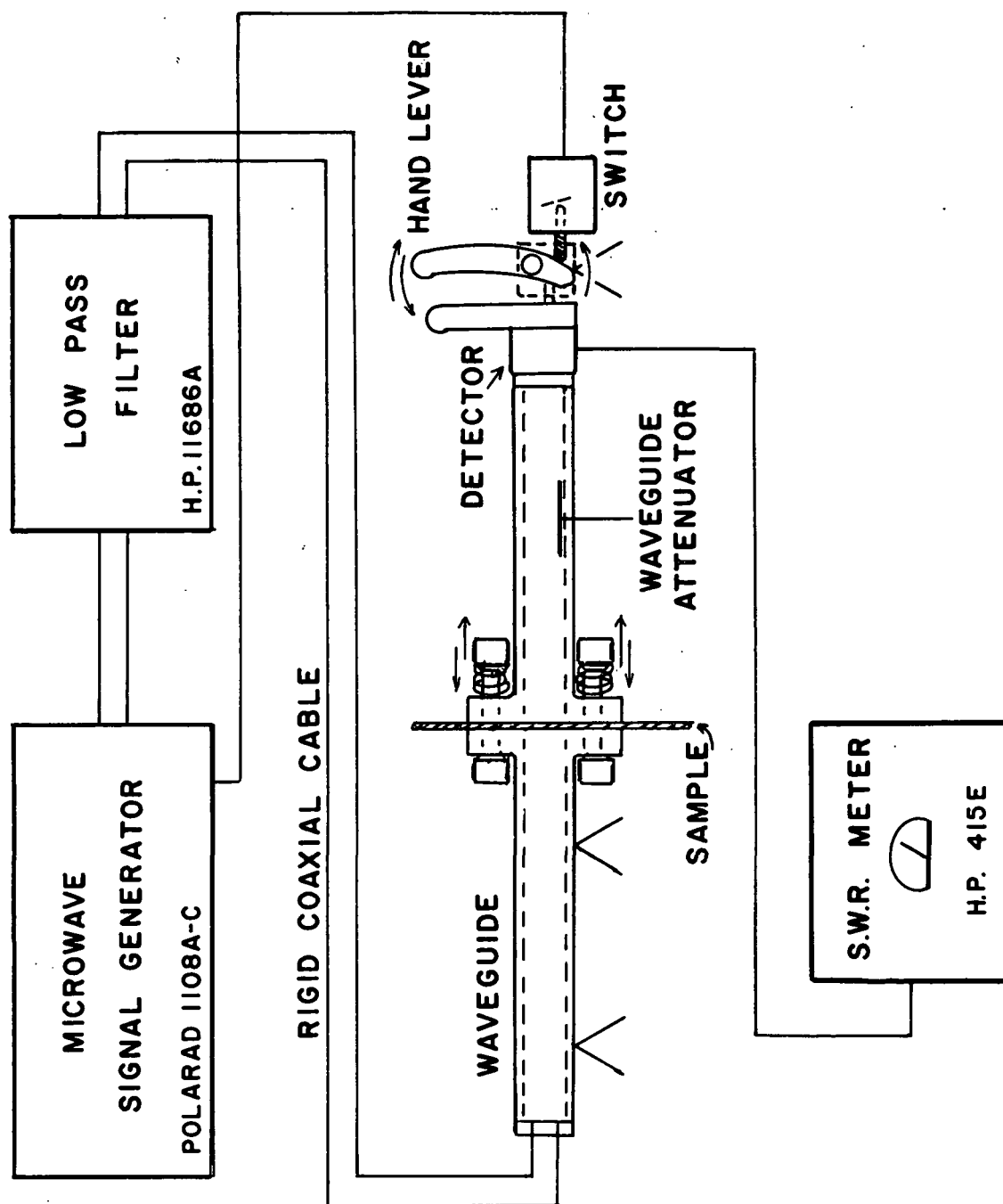


Figure 1. Schematic diagram of the microwave fiber orientation gage.

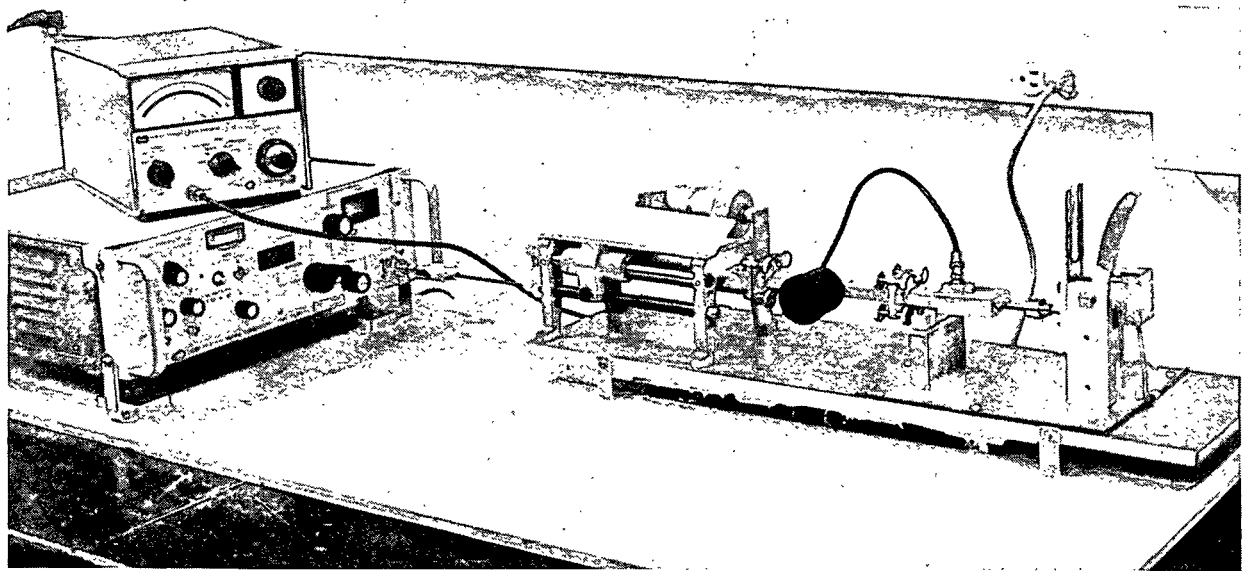


Figure 2. Photograph of the experimental apparatus.

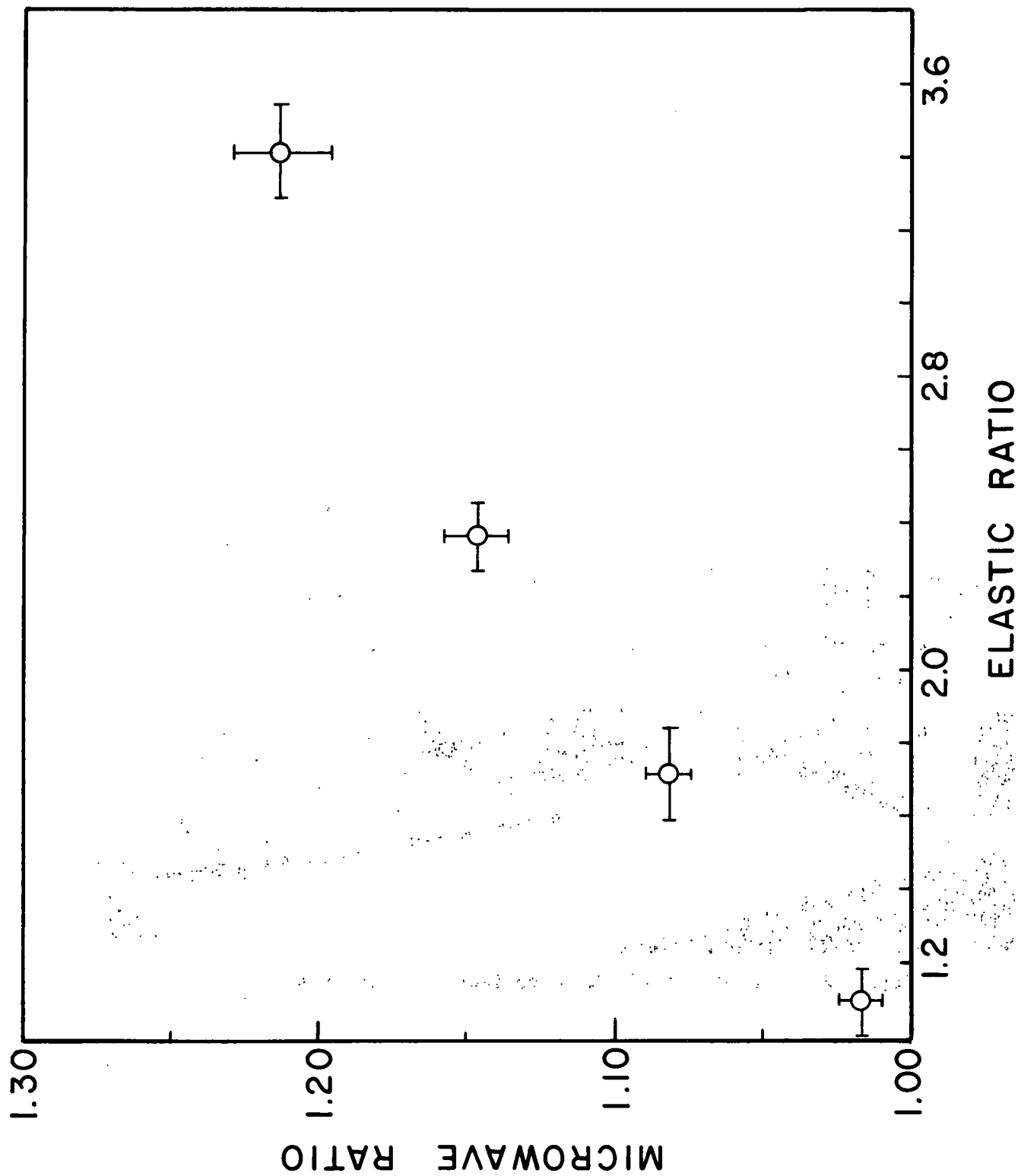


Figure 3. Microwave anisotropy ratio vs. elastic anisotropy ratio for bleached kraft, Douglas fir sheets made to different fiber orientation distributions.

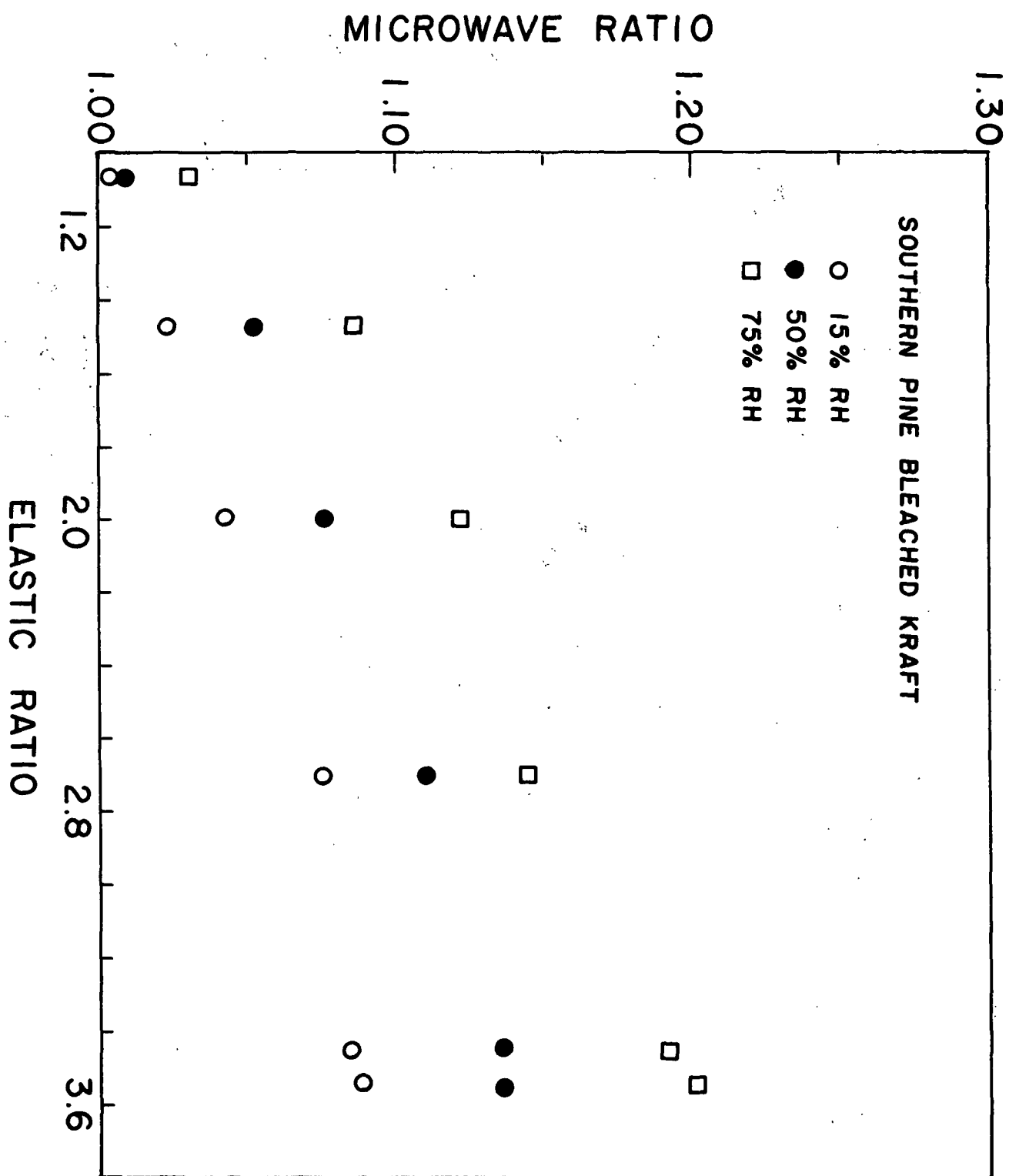


Figure 5. The effect of moisture on the microwave anisotropy ratio.

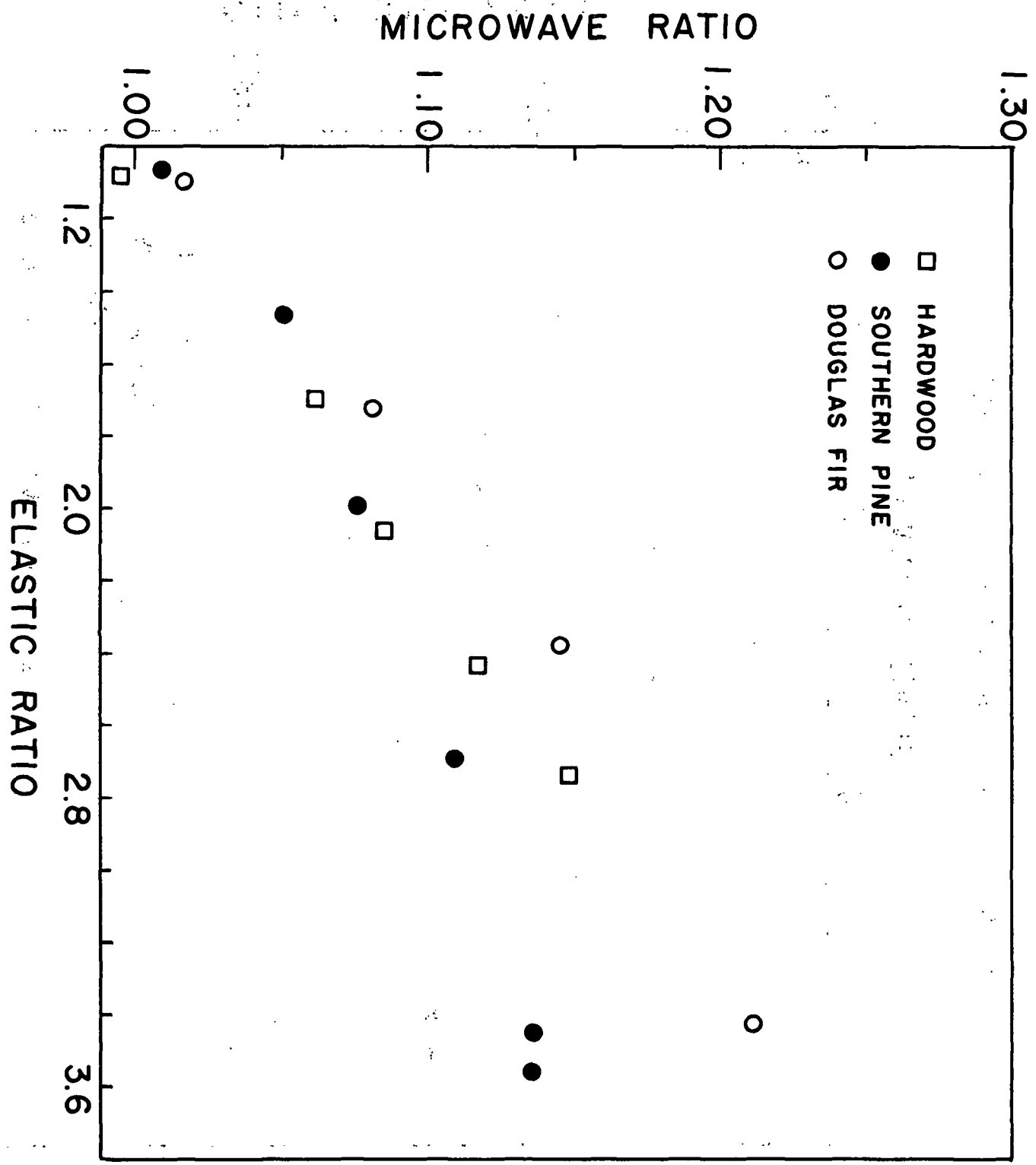


Figure 4. The effect of varying furnish on the relationship between microwave anisotropy ratio and elastic anisotropy ratio.

IPC TECHNICAL PAPER SERIES #166

AUTHOR

Use of Microwave Attenuation as

TITLE a Measure of Fiber Orientation Anisotropy

IPC TECHNICAL PAPER SERIES #166

**GEORGIA-PACIFIC
CORPORATION
ATLANTA, GEORGIA**